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(57)【特許請求の範囲】

(57)[CLAIMS]

【請求項１】
成長させるべき単結晶の原料を収容する坩堝と、前記原料を加熱溶融する加熱手段と、前記坩堝内の融液から単結晶を成長させつつ引上げる手段とを具備する単結晶成長装置において、前記坩堝内の融液の上方であって、且つ単結晶の引上げ域の周囲に配設され、上側から下側に向かうに従って縮径された筒状の金属製遮閉部材と、該金属製遮閉部材に付設された冷却手段と、前記金属製遮閉部材の外周面を溶融原料からの蒸発物及び輻射熱源から遮閉すべく前記外周面の外側に所要の間隙を隔てて配設された筒状の遮閉部材とを具備することを特徴とする単結晶成長装置。

[CLAIM 1]
In the single-crystal growth apparatus which to comprise a melting pot which accommodates a raw material of a single crystal to grow, heat means which carries out heat-melting of the above-mentioned raw material and means which pulls up, growing up a single crystal from the melt solution in the above-mentioned melting pot, a cylindrical metal shield member reduced to the bottom from the upper side which is arranged in the upper part of the melt solution in the above-mentioned melting pot, and the perimeter of the pulling-up region of a single crystal, a cooling means attached to this metal shield member, and a cylindrical shield member arranged in the outside of the above-mentioned outer circumferential surface by separating a required interstice to shield the outer circumferential surface of the above-mentioned metal shield member from the emission from a melting raw material and the

source of the radiant heat are comprised.

The single-crystal growth apparatus characterised by the above-mentioned.

【請求項２】

坩堝内の融液の上方であって、且つ単結晶の引上げ域の周囲に配設され、上側から下側に向かうに従って縮径された筒状の金属製遮閉部材の外周面の外側に所要の間隙を隔てて配設された筒状の遮閉部材が黒鉛製であることを特徴とする特許請求の範囲第１項記載の単結晶成長装置。

[CLAIM 2]

The cylindrical metal shield member reduced to the bottom from the upper side which is arranged in the upper part of the melt solution in the above-mentioned melting pot, and the perimeter of the pulling-up region of a single crystals made from graphite.

The single-crystal growth apparatus described in the 1st claim characterised by the above-mentioned.

【発明の詳細な説明】

[DETAILED DESCRIPTION OF INVENTION]

【産業上の利用分野】

本発明はチョクラルスキー法による単結晶成長装置に関するものである。

[INDUSTRIAL APPLICATION]

This invention relates to the single-crystal growth apparatus by the Czochralski method.

【従来技術】

第３図は一般的なチョクラルスキー法（CZ 法）による単結晶成長装置の要部の模式的断面図である。１は図示しないチャンバ内に配した坩堝であってグラファイト製の容器 1a の内側に石英製の容器 1b を配した二重構造に構成され、その下底部中央にはこれを回転、並びに昇降移動させる軸 1c が連結されている。また坩堝１の周囲には円

[PRIOR ART]

Third diagram is a typical sectional drawing of the principal part of the single-crystal growth apparatus by the general Czochralski method (CZ process).

1 is the melting pot distributed in the chamber not to be illustrated. It is constituted of the double structure that a vessel 1b made of quartz is arranged in the inside of the vessel 1a made of graphite.

A shaft 1c which rotates and moves upward and downward this is connected with the center

筒形をなす発熱体２が、更に坩堝１の上方には種結晶３に成長させた単結晶４を引上げる引上手段（図示せず）が配設されている。

而して、このような結晶成長装置にあっては坩堝１内に収容した原料を発熱体２により加熱溶融せしめ、その融液に浸した種結晶３を引上手段にて回転しつつ上昇させ、種結晶３の下端に単結晶４を成長せしめるようになっている。

一般に単結晶の引上げを行う場合、その引上げ速度は、引上げ方向における単結晶の温度勾配と密接な関係にあり、効率的な単結晶の引上げを行うには単結晶を所定以上の温度勾配に設定する必要がある。ところで上記した如き装置では単結晶４の周囲には坩堝１、発熱体２及び融液等の輻射熱源があってこれらから受ける輻射熱量が極めて大きく、必然的に単結晶４の引上方向における温度勾配が小さくなり、単結晶の引上げ効率が低いという欠点があった。

この対策として従来にあっては第４図に示す如く坩堝１の上方であって単結晶４の引上げ域の周囲に、扁平な環状リム 7a とその内側端縁から下方に向かうに従って縮径した円錐台形のテーパ部 7b とからなる輻射スクリーン７を配設する構成が提

of the lower bottom-part.

Moreover a tubular heat-emitting body 2 is arranged in the perimeter of the melting pot 1. Furthermore, the pulling-up means (not shown) to pull up the single crystal 4 grown up into the seed crystal 3 is arranged in the upper part of the melting pot 1.

Thus, in such a crystal growth apparatus, heat melting of the raw material accommodated in the melting pot 1 is carried out by the heat-emitting body 2. The seed crystal 3 dipped in the melt solution is pulled up with the pulling-up means, rotating it.

A single crystal 4 is grown at the lower end of a seed crystal 3.

Generally, when pulling-up a single crystal, the pulling-up velocity is in a relationship closely with the temperature gradient of the single crystal in the pulling-up direction.

For efficiently pulling-up a single crystal, a single crystal needs to be set as the temperature gradient more than a predetermined.

Incidentally, in the apparatus which was described above, there are sources of a radiant heat, such as a melting pot 1, a heat-emitting body 2, and a melt solution, in the perimeter of a single crystal 4. The amount of radiant heats received from these is very large.

The temperature gradient in the pulling-up direction of a single crystal 4 becomes small inevitably.

There was a fault that the pulling-up efficiency of a single crystal was low.

As this countermeasure, conventionally, the constitution which arranges the radiation screen

案されている（特公昭57-40119号）。

この輻射スクリーン7によって坩堝1、発熱体2及び融液等からの輻射熱を遮断し、単結晶4の引上方向における温度勾配を高め、また同時にチャンバの上方から坩堝1に向けて給送されるAr等のキャリアガスを坩堝1内に誘導し、坩堝1から生成されるSiOガス等を坩堝1の下方に導き、ここからチャンバの外部に排出するようにしてある。

【発明が解決しようとする問題点】

しかし上述した如き従来の装置にあっては輻射スクリーン7によって坩堝1、発熱体2、融液等と単結晶4との間が遮断される結果、これらからの一次的輻射熱は遮断されるものの、これによって輻射スクリーン7自体も高温に加熱される結果、二次輻射熱が単結晶4に向けて放射され、単結晶4自体の冷却効果が十分でなく、また輻射スクリーン7自体は金属で形成されるが、融液から発生するSiOガス、

7 which consists of a flat cyclic rim 7a and a taper part 7b of a cone trapezoid reduced from the inner-side edge to the lower part, in the upper part of a melting pot 1 and the perimeter of the pulling-up region of a single crystal 4 as shown in Diagram 4 is proposed (Japanese Patent Publication No. 57-40119).

The radiant heat from the melting pot 1, the heat-emitting body 2, and the melt solution, etc. is interrupted with this radiation screen 7.

The temperature gradient in the pulling-up direction of a single crystal 4 is raised.

Moreover a carrier gas like Ar which is fed toward the melting pot 1 from the upper part of a chamber, is guided into the melting pot 1 simultaneously. SiO gas formed from the melting pot 1 is guided underneath the melting pot 1.

It is made to eject from here into the outside of the chamber.

[PROBLEM ADDRESSED]

However in the above-mentioned conventional apparatus, Between a melting pot 1, the heat-emitting body 2, the melt solution, and the single crystals 4 is interrupted with the radiation screen 7.

As a result, the primary radiant heat from these is interrupted. However, the radiation screen 7 itself is also heated by this to a high temperature.

As a result, a secondary radiant heat is radiated toward the single crystal 4.

Cooling effect of a single-crystal 4 itself is not enough. Moreover the radiation screen 7 itself is formed with metal.

或いは高温のために溶融劣化されて寿命が短く、溶融劣化物が坩堝１内の融液中に落下して融液を汚染し、また落下物自体が多結晶化を誘引する等の問題があった。

本発明はかかる事情に鑑みなされたものであって、その目的とするところは単結晶にその引上げ方向に効果的に温度勾配を形成し、単結晶の引上げ速度を高め、また坩堝内への異物の落下混入を防止し得るようにした単結晶成長装置を提供するにある。

【問題点を解決するための手段】

本発明装置にあっては冷却手段を付設した金属製遮閉部材と該金属製遮閉部材の外周にこれとの間に間隔を隔てて配設された黒鉛製遮閉部材とを具備する。

【作用】

これによって単結晶の引上げ方向における温度勾配を高め得て結晶引上げ速度を高く維持し得る。

又、成長方向での OSF,酸素析出等の品質を向上し得る。

【実施例】

However, because of SiO gas which emits from the melt solution, or the high temperature, melting degradation is carried out and its durability is short.

The melting degradation substance drops in the melt solution in the melting pot 1 and then the melt solution is contaminated.

Moreover there was a problem of a drop substance itself inducing a polycrystalisation.

This invention is made in view of such a situation.

The objective is to provide a single-crystal growth apparatus which forms a temperature gradient in the pulling-up direction effectively to a single crystal, and raises the pulling-up velocity of a single crystal, and prevents the foreign material from dropping and mixing into a melting pot.

[SOLUTION OF PROBLEMS]

In this invention apparatus, it comprises a metal shield member which attached a cooling means and a shield member made of graphite which is arranged between this and the periphery of this metal shield member by separating a interval.

[Effect]

This can raise the temperature gradient in the pulling-up direction of a single crystal, and keeps the crystal pulling-up velocity high.

Moreover, quality, such as OSF in the growth direction and oxygen precipitate, may be improved.

[Example]

以下本発明をその実施例を示す図面に基づき具体的に説明する。第１図は本発明に係る単結晶成長装置（以下本発明装置という）の模式的断面図であり、図中１は坩堝、２は発熱体、３は種結晶、４は単結晶、８は金属製遮閉部材、９は黒鉛製遮閉部材を示している。坩堝１は黒鉛製の容器 1a の内側に石英製の容器 1b を配設した二重構造に構成されており、その下底部中央には坩堝１を回転、並びに昇降させる軸 1c が連結されている。また坩堝１の側周にはこれとの間に所要の間隔を隔てて円筒形の発熱体２が配設され、また、坩堝１の上方には、種結晶３及びこれに単結晶４を成長させつつ引上げる引上装置（図示せず）が配設されている。

遮閉部材８は銅等の金属製であって、上，下端が開放され、上端から下端に向かうに従って内，外径ともに縮径された中空の逆円錐台形に形成され、坩堝１内の融液の直上であって、単結晶４の引上げ域の周囲にこれと同心の状態で、且つ大径端を上方に、小径端を下方にして配設されており、その内周面にはこれに沿わせて冷却手段を構成する冷却水の冷却水管 8a が螺旋形に配設されている。

一方遮閉部材９は黒鉛製であって、遮閉部材８よりも若干大

This invention is concretely demonstrated below based on the drawings which show the Example. Diagram 1 is a typical sectional drawing of the single-crystal growth apparatus (henceforth it is called this invention apparatus) based on this invention. 1 is a melting pot in the drawing(s). 2 is a heat-emitting body. 3 is a seed crystal. 4 is a single crystal. 8 is a metal shield member. 9 shows the shield member made of graphite. The melting pot 1 is constituted by the double structure which arranges a vessel 1b made of quartz in the inside of a vessel 1a made of graphite. A shaft 1c which makes a melting pot 1 rotate and elevate is connected with the center of the lower bottom-part. Moreover in the side periphery of the melting pot 1, the cylindrical heat-emitting body 2 is arranged a required interval apart from this. Moreover, the seed crystal 3 and the pulling-up apparatus (not shown) which pulls up, growing up a single crystal 4 into this is arranged in the upper part of the melting pot 1.

The shield member 8 etc. is made of metal like copper. The upper and lower ends are is open. It forms a hollow reverse cone trapezoid by which the inner and outer diameters were reduced toward the lower end from the upper end. It is arranged just above the melt solution in the melting pot 1, in the concentric condition with this around the pulling-up region of a single crystal 4, making the larger diameter end upward and the smaller diameter end downward. A cooling-water pipe 8a of the cooling water which constitutes a cooling means along with this is arranged in the

きい相似形をなす中空の逆円錐台形に形成され、遮閉部材８の外側に外周面との間に排気用通気路を構成する所要の間隙を隔てて同心状に配設されている。

而してこのような本発明装置にあっては坩堝１、発熱体２等の輻射熱源からの熱は黒鉛製遮閉部材９、冷却水管 8a を付設された金属製遮閉部材８によって遮断し、また単結晶４と対向する金属製遮閉部材８は強制冷却される結果、金属製遮閉部材８が高温に加熱されることがなく、従って単結晶４に対する輻射熱源として作用することがない。しかも黒鉛製遮閉部材９は金属製遮閉部材８の外周を覆って坩堝１から生ずるSiO,CO,CO2 ガスを遮断するから金属製遮閉部材８の劣化或いは溶解も防止することが出来ることとなる。

また単結晶４の引上げ過程でチャンバの上方から坩堝１の上部に向けて供給される Ar ガス等のキャリアガスは金属製遮閉部材８に沿って、或いはこれと黒鉛製遮閉部材９との間の間隙に沿って坩堝１内の融液面の中央に導かれ、ここから坩堝１の周縁部に流れて発生したSiO ガス等と共に坩堝１の周縁部から外方に流出し、坩堝１と発熱体２との間の通気路を経て坩堝１の下方に流下し、図示しない排

internal-circumference surface by a spiral form.

On the one hand, the shield member 9 is made of graphite. It forms a hollow reverse cone trapezoid which has a similar type a little larger than the shield member 8. It is arranged in the outside of the shield member 8 concentrically between the outer circumferential surfaces, by separating by a required interstice by which constitutes a ventilator for an exhaust gas.

Thus, in such this invention apparatus, the heat from the sources of a radiant heat like the melting pot 1 and the heat-emitting body 2, is interrupted by the metal shield member 8 attached to the shield member made of graphite 9 and cooling-water pipe 8a. Moreover the metal shield member 8 opposite to the single crystal 4 is forcedly cooled down. As a result, the metal shield member 8 is not heated to a high temperature, therefore it does not work as a source of a radiant heat opposed to a single crystal 4. And since the graphite manufacturing shield member 9 covers the periphery of the metal shield member 8 and interrupts SiO which is produced from the melting pot 1, CO, and CO2 gas, it can also prevent degradation or melt of the metal shield member 8.

Moreover carrier gas like Ar gas supplied toward the upper part of a melting pot 1 from the upper part of a chamber in the pulling-up process of a single crystal 4, is guided to the center of the melt-solution surface in the melting pot 1, along with the metal shield member 8 or along with the interstice between this and the graphite manufacturing shield

気口に導かれることとなって、キャリアガスの誘導、SiO ガス等の排出に何らの支障も生じることはない。

次に本発明装置についての試験結果を具体的数値を掲げて説明する。

坩堝１の融液面上に下端部内径 :200mm 、 上 端 部 内径:400mm、高さ:300mm の銅製遮閉部材８をその下端を融液上10mm に位置させて配設し、内周面に配設した冷却水管 8a には 30l/分の冷却水を通流させた。またその外周に 10mm の間隙を隔てて黒鉛製遮閉部材９を配設し、直径５インチの単結晶成長を行った。その結果単結晶引上げ速度は 2.5mm/分を達成し得た。

なお、これによって得た単結晶は成長方向が均一で酸化熱処理（1000℃×16 時間）に依っても表面の積層欠陥、内部微小欠陥を生じることがなかった。また発熱体２のための使用電力は30%低減することが出来た。

第２図は本発明装置と従来装置との単結晶成長速度の比較試験結果を示すグラフであり、横軸に引上率（%）を、また縦軸に成長速度（mm/分）をとって示してあり、グラフ中実線は本発明装置の、また破線は第３図に示す従来装置の各結果を示している。このグラフから明らか

member 9. It flows into the circumference part of the melting pot 1 from here. It flows into outside out of the circumference part of a melting pot 1 with SiO gas which generated. It flows underneath the melting pot 1 through the ventilator between the melting pot 1 and the heat-emitting body 2. It guides to the exhaust port not to illustrated. Not any trouble occurs in introduction of carrier gas or emission of SiO gas, etc.

Next referring to concrete numerical values, the test result about this invention apparatus is demonstrated.

The shield member (lower-end part internal-diameter:200 mm, upper-end part internal-diameter:400 mm, and height:300 mm) made from copper 8 is arranged on the melt-solution surface of the melting pot 1, positioning the lower end at 10 mm over the melt solution. The cooling water of 30l/min pass through the cooling-water pipe 8a arranged in the internal-circumference surface. Moreover the graphite manufacturing shield member 9 is arranged in the periphery, by separating by 10 mm interstice. The single-crystal growth with a diameter of 5 inches was performed. As a result, single crystal pulling-up velocity could attain 2.5 mm/min.

In addition, the single crystal obtained by this has the uniform growth direction. Even the heat-of-combustion process (1000 degree-C * 16 hours) does not produce the surface stacking fault and the internal micro-defect were not produced. Moreover the used electric power for a heat-emitting body 2 was able to be reduced by 30%.

な如く従来装置では結晶成長速度が引上率の上昇と共に、漸次低下するのに比較して本発明装置に依れば引上率の如何にかかわらず終始一貫した単結晶成長速度を維持し得ていることが解る。

なお上述の実施例では冷却手段を構成する冷却水管 8a は金属製遮閉部材８の内周面に螺旋形に配設する構成を示したが、何らこれに限らず金属製遮閉部材８内にこれと一体的に配設してもよい。

The second diagram is a diagrammatic chart in which the comparison test result of the single-crystal growth rate of this invention apparatus and the conventional apparatus is shown. It is shown by the horizontal axis of pulling-up percentage (%) and the vertical axis of the growth rate (mm/min). In the diagrammatic chart, Each sequence of this invention apparatus with a continuous line and the conventional apparatus shown in Third diagram with a broken line is shown. It is proved clear from this diagrammatic chart that while in the conventional apparatus, the crystal growth rate reduces gradually with a raise of the pulling-up rate, according to this invention apparatus, the consistent single-crystal growth rate can be completely maintained, irrespective of the pulling-up rate.

In addition, the constitution is showed in the above-mentioned Example that the cooling-water pipe 8a which constitutes the cooling means is arranged with a spiral form in the internal-circumference surface of the metal shield member 8.

However, it may not restrict to this at all, but it may arrange integrally with this in the metal shield member 8.

【効果】

以上の如く本発明装置にあっては冷却手段を備えた金属製遮閉部材、黒鉛製遮閉部材によって坩堝等の輻射熱源からの輻射熱を遮断するから単結晶の引上方向における温度勾配を適正に保持し得て単結晶の引上速度を終

[Effect]

As mentioned above, in this invention apparatus, since the radiant heat from the sources of a radiant heat, such as a melting pot is interrupted by the metal shield member equipped with cooling means and the graphite manufacturing shield member, the temperature gradient in the pulling-up direction of a single

始一定に維持し得ることとなり、また金属製遮閉部材それ自体には冷却手段が設けられているため、単結晶と対向する金属製遮閉部材自体から受ける輻射熱は少なく、成長方向での均一性も良くなる。更に坩堝と対向する側には黒鉛製遮閉部材を設けてあるから坩堝から発生する蒸気による金属の劣化を防止し得、金属製遮閉部材の寿命延長、並びにこれの使用に伴う弊害を除去出来ることとなって安定した高効率の結晶成長を行い得るなど本発明は優れた効果を奏するものである。

crystal can be kept appropriate. The pulling-up velocity of a single crystal can be kept uniformly from beginning to end.

Moreover since cooling means is provided to metal shield member itself, the radiant heat received from the metal shield member itself opposed to a single crystal is little, and the uniformity in the growth direction also becomes good.

Furthermore since the graphite manufacturing shield member is provided to the side opposed to a melting pot, metal degradation by the vapour which generates from the melting pot can be prevented.

The life extension of a metal shield member and the bad effect accompanied by usage of this is removable. Therefore, stable crystal growth with high-efficiency can be performed. This invention has such an outstanding effect.

【図面の簡単な説明】

第１図は本発明装置の模式的断面図、第２図は本発明装置と従来装置との結晶成長速度の比較試験結果を示すグラフ、第３図はチョクラルスキー法の一般的な構成を示す模式的断面図、第４図は従来装置の模式的断面図である。

1……坩堝、1a……黒鉛製容器
1b……石英製容器、1c……軸、
2……発熱体
3……種結晶、４……単結晶、
8……金属製遮閉部材
9……黒鉛製遮閉部材

[BRIEF EXPLANATION OF DRAWINGS]

Diagram 1 is a typical sectional drawing of this invention apparatus. The second diagram is a diagrammatic chart in which the comparison test result of the crystal growth rate of this invention apparatus and the conventional apparatus is shown. The 3rd diagram is a typical sectional drawing showing the general constitution of a Czochralski method. The diagram 4 is a typical sectional drawing of a conventional apparatus.

1..... melting pot, 1a..... graphite manufacturing vessel

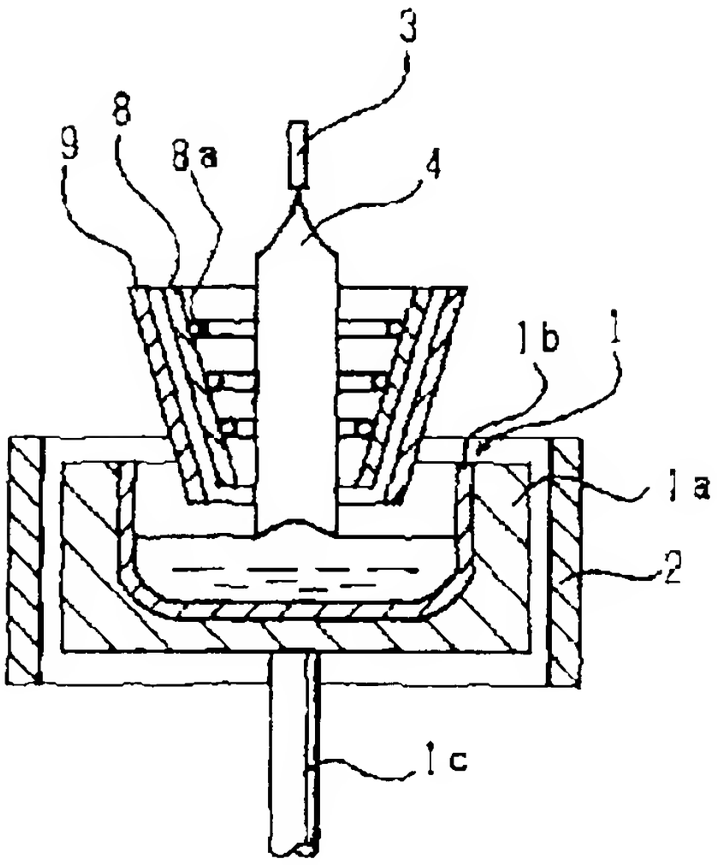
1b..... quartz manufacturing vessel, 1c..... shaft, 2..... heat-emitting body

3..... seed crystal, 4..... single crystal, 8.....

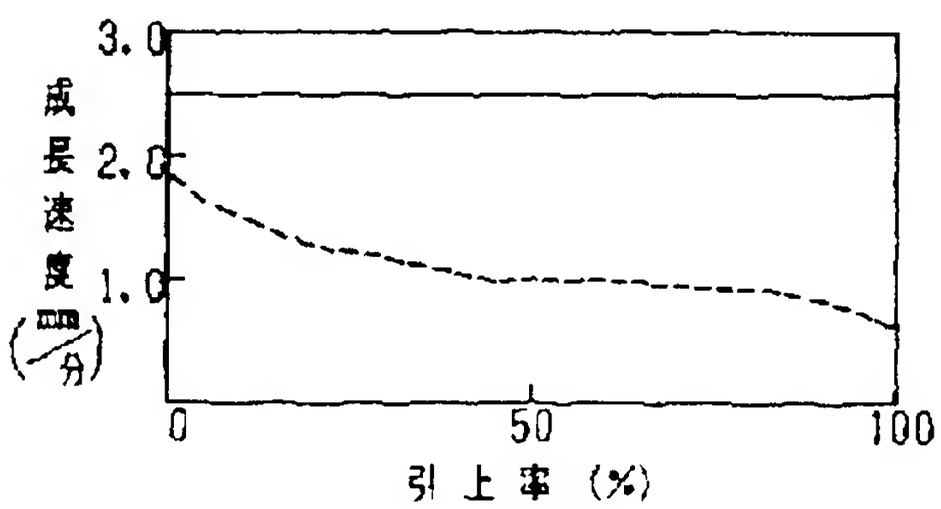
metal shield member

9..... shield member made of graphite

【第１図】 **[A diagram 1]**



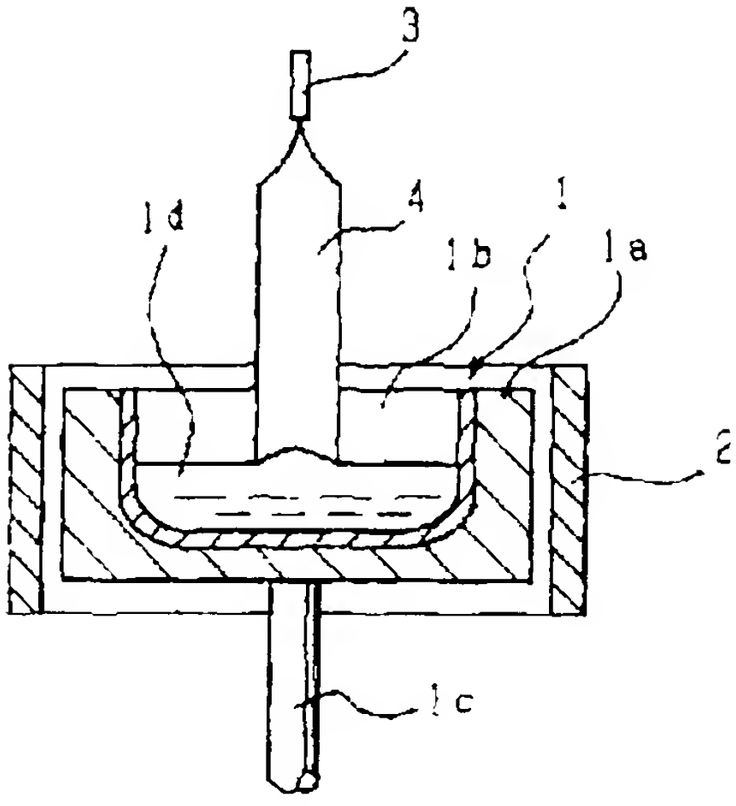
【第２図】 **[A second diagram]**



Vertical axis : Growth rate

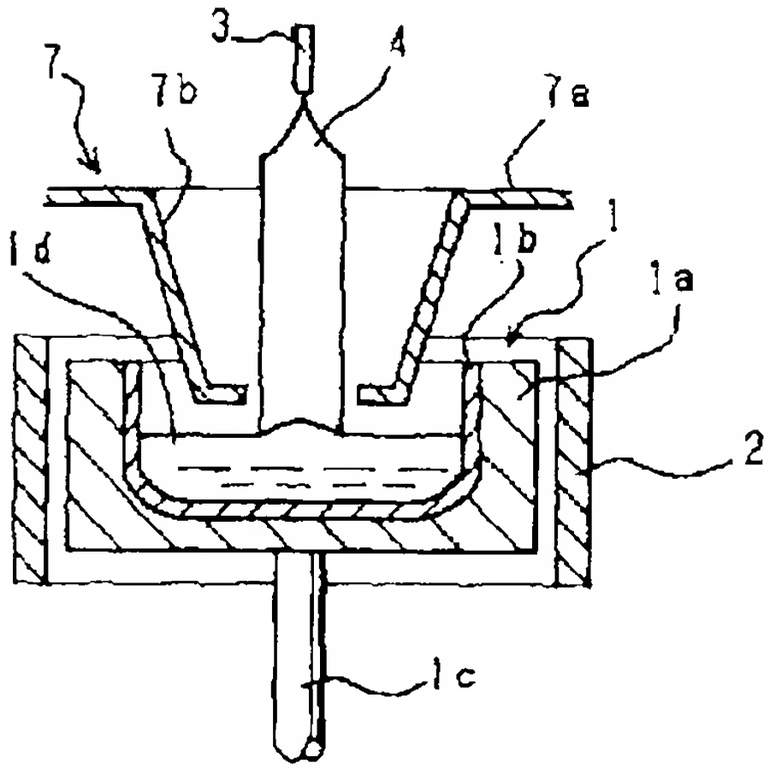
Horizontal axis : Pulling-up rate

【第３図】 **[A 3rd diagram]**



【第４図】

[A diagram 4]



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